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Statistical Analysis of Grain Structure Features in Ni-Cu Alloys at the Saturation Stage Under High-Pressure Torsion

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Abstract. Statistical analysis of the Ni-Cu system grain structure formed at room temperature under high-pressure torsion at steady-state deformation (saturation stage) is done. It is demonstrated that, in all the alloys considered (with 10, 34, and 90 at.% of Cu) there are two groups of crystallites, in one of which a pronounced effect of relaxation processes is observed, whereas they are not revealed in the other one. The ratio of the volume fractions of these groups depends on the alloy composition and, correspondingly, on its melting temperature. It is shown that the final average grain size is formed under the effect of the dominating crystallite group depending on the alloy melting temperature.

INTRODUCTION

In the last decades, bulk metallic materials subjected to severe plastic deformation (SPD) with ultrafine-grained (UFG) structure attract great attention of researchers due to their unique strength, plastic, diffusion, and other characteristics [1, 2].

One of the most effective techniques of SPD is high pressure torsion (HPT) [3]. At the same time, the nanostructured state, namely the structure with prevailing crystallites less than 100 nm in size with high-angle boundaries, can by no means always be prepared. The main cause consists in reaching the saturation stage characterized by the absence of further structural refinement and material hardening with an increasing degree of deformation [4, 5]. At the saturation stage, equilibrium is set between the generation and annihilation of defects (vacancies, dislocations, and high- and low-angle boundaries). The main process that limits the possible of refinement is grain-boundary migration. The minimum grain sizes obtained by HPT are determined by, first of all, the nature of the deformed material, in particular, its crystal lattice, melting temperature, and stacking fault energy (SFE) [4, 6].

Copper and nickel are fairly plastic materials, and they can be deformed to high strains without failure at relatively low temperatures; therefore, the structure and properties of these metals subjected to SPD were studied in many publications [7–15]. The authors pointed out a significant temperature influence on the formation possibility of a nanocrystalline structure [9–11]. Another factor, which substantially affects structure formation upon SPD, is the stacking fault energy [6, 16]. In materials with high SFE, dislocations and dislocation sliding play a dominant role [17, 18].

Along with the UFG structure, there is the presence of so-called nonequilibrium crystallite boundaries, characterized by excess energy, long-range elastic stresses, and higher free volume [19]. In a number of studies, it was confirmed that the grain boundary state in such materials substantially differs from the state of recrystallization-originated boundaries in coarse-grained polycrystalline [20–24].

In the recent works [25, 26], a method based on the statistical analysis of grain size distributions in materials subjected to SPD was proposed. This method allows grains to be divided into different groups based on a suggestion that there exist more than one group of grains with common characteristics. It was shown that this division can be carried out by fitting several distributions with subsequent analysis of the calculated parameters of distributions such

as average grain size, standard deviation, and volume fraction. It allows one to perform deep analysis of the behavior of crystallite groups, with heating or changing a chemical composition.

The aim of this study is to develop the existing data of Cu-Ni alloys [27, 28] based on the analysis of the structure formed at the saturation stage.

RESULTS AND DISCUSSION

According to the studies [29–32], the fragmentation of the structure upon deformation occurs as follows. As the degree of deformation increases, dislocations are accumulated and localized at cell walls; the misorientation of the cells increases. This leads to the formation of a UFG structure. The further deformation activates rotation deformation modes over the sample volume and ensures the occurrence of the saturation stage when the dislocation mobility is the determining factor for structure fragmentation. The dislocation mobility can be decreased in two ways. One way is a decrease in the SFE (in particular, by alloying) [27, 28], and the other is a decrease in the deformation temperature (or a decrease in the homologous temperature in the case of different materials) [10, 11, 14].

Earlier [27], the formation of a structure at the saturation stage in Cu-Ni system was considered, but the detailed analysis of the structures has yet to be carried out. At the same time, in [25, 26], the possibility of dividing grain structure into different groups taking into account specific features of distributions in every group was demonstrated. The use of the calculated parameters of grain size distributions was proposed to identify groups of grains by their origin. It was shown that the grain structure of nanostructured materials can have several groups of grains with various behaviors during further heating. Consequently, a different state of grain boundaries for these crystallite groups is supposed, since the grain-boundary migration determines the behavior of crystallites during further heating. In addition, it was shown in [3] that, at the saturation stage, the grain structure relaxation mechanism can be directly realized in the deformation process by moving the already formed grain boundaries.

The data reported in [27] is taken for the analysis. The division into different groups of grains was performed by the approach worked out in [26]. Crystallite groups were approximated by the model proposed in [25].

The results of the fitting are presented in Fig. 1. The division of grains into groups according to the characteristics of their distributions is carried out based on the principle which suggested subsequent analysis of the calculated distributions parameters, such as average grain size, standard deviation, and volume fraction.

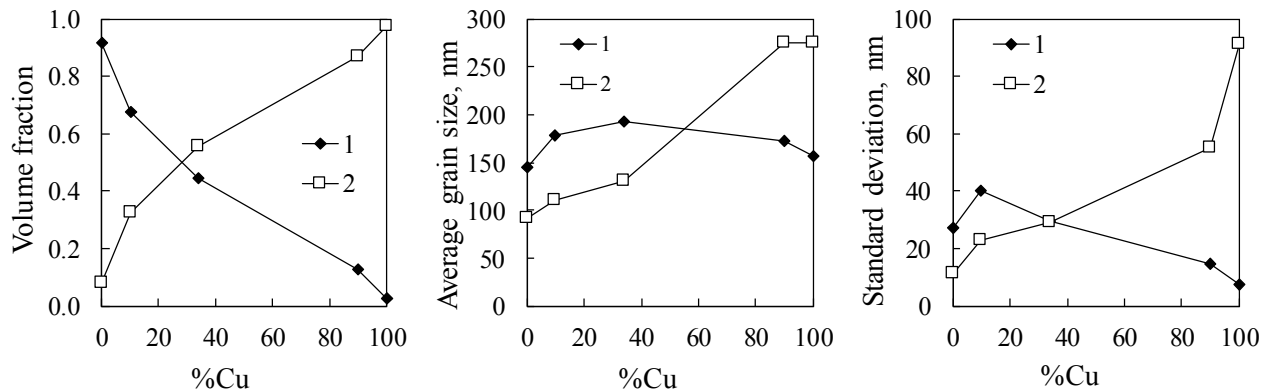


FIGURE 1. Approximation results obtained from processing the grain size distribution histograms of various Ni-Cu compositions

It can be seen that the experimental distribution can be decomposed into two separate distributions with larger and smaller average grain sizes, which also possess different values of standard deviation. This fact indicates accurate distinction between these groups of grains.

The volume fraction changes of grain groups indicate that, at the point corresponding to pure Ni, group 1 with the average grain size of 145 nm dominates, and minor group 2 has an average gain size of about 90 nm. At the same time, the dominance of group 2 is observed at the point of pure Cu, where the average grain size of group 1 is 157 nm and that of group 2 is 274 nm.

This can be explained by the tendency of the alloys to undergo relaxation processes. Since, in contrast to pure Cu, where the influence of relaxation processes is shown [14], Ni has a significantly higher melting point, and the relaxation process, as a rule, is not observed during and after deformation [10–13]. Thus, the group of crystallites which has not undergone relaxation processes will dominate in pure Ni. In pure Cu, the group of grains formed with the influence of relaxation processes dominates, this being consistent with the data obtained in [7, 14, 15]. Accordingly, group 1 can be formed by crystallites with grain boundaries of the deformation origin, where relaxation processes only begin with temperature increase, and group 2 can include crystallites in which relaxation processes have already taken place.

Moreover, the ratio of volume fractions of these groups depends on the alloy composition and, correspondingly, on its melting temperature. There is every indication that the final average grain size is formed under the effect of the dominating crystallite group depending on the alloy melting temperature.

In addition, it should be noted that the non-linearity in the crystallite sizes and volume fractions of crystallite groups depending on the alloy composition is observed. As proposed earlier in [27, 28], this is likely due to the substantial effect of variations in the stacking-fault energy with a changing composition. Thus, the observed role of the stacking-fault energy in the formation of the alloy structure is an important factor, which should be taken into account.

CONCLUSION

Statistical analysis of the Ni-Cu grain structure formed at room temperature under high-pressure torsion at steady-state deformation (saturation stage) has been done.

It has been demonstrated that in all the alloys considered (with 10, 34, and 90 at.% of Cu) there are two groups of crystallites, in one of which a pronounced effect of relaxation processes is observed, whereas they are not revealed in the other one. The ratio of the volume fractions of these groups depends on the alloy composition and, correspondingly, on its melting temperature. It has been demonstrated that the final average grain size is formed under the effect of the dominating crystallite group depending on the alloy melting temperature.

The non-linearity in the crystallite sizes and volume fractions of crystallite groups depending on the alloy composition is observed, indicating the noticeable effect of the stacking-fault energy on the structure forming under deformation.

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